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BULLETIN NO. 8

South Dakota School of Mines

Departments of Geology, Chemistry, Mining and Metallurgy.

UNIVERSITY OF ILLINOIS
PRESIDENT'S OFFICE.

THE

Cement Resources

OF THE

Black Hills.

Including Tests on
Cements Made from Black Hills Material.

BY

CLEOPHAS C. O'HARRA, Ph. D.
M. F. COOLBAUGH, M. A.
M. A. EHLE, Jr. E. M.
CHAS. H. FULTON, E. M.

RAPID CITY, S. D.
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RAPID CITY, S. D.
JULY, 1908.

Rapid City Printing Company,
Rapid City, S. D.

Letters of Transmittal.

SOUTH DAKOTA SCHOOL OF MINES.

Rapid City , S. D., July 1, 1908.

Dear Sir:—

I have the honor to transmit herewith a paper by C. C. O'Harra, M. F. Coolbaugh, Mark Ehle and C. H. Fulton on the Cement Resources of the Black Hills of South Dakota. I submit it with the recommendation that it be published as Bulletin No. 8 of the School of Mines.

Respectfully,

CHARLES H. FULTON,

President.

To The Hon. E. C. Ericson, President,

Regents of Education.

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Introduction.

The great increase in the use of Portland Cement has drawn attention to possible sources of raw material. The Black Hills of South Dakota are especially favored in this respect, both as regards the quantity available, and the purity of the material. In the experimental work on the cements care was taken to have it thorough and representative. Much more work was done than is recorded in the bulletin.

The editor desires to express thanks to Mr. William A. Coursen for the efficient aid given in the construction and operation of the experimental cement furnace; also to thank Mr. J. B. Read for services in connection with the making of analysis, etc. The editor also expresses his gratitude to his colleagues, Professors O'Harra, Coolbaugh and Ehle for their able labors in making this bulletin thorough and representative.

CHARLES H. FULTON,
Editor.

PART I.

THE GEOLOGY OF THE BLACK HILLS CEMENT MATERIAL.

C. C. O'HARRA, Ph. D.

The Geology of Black Hills Cement Material.

C. C. O'HARRA, PH. D.

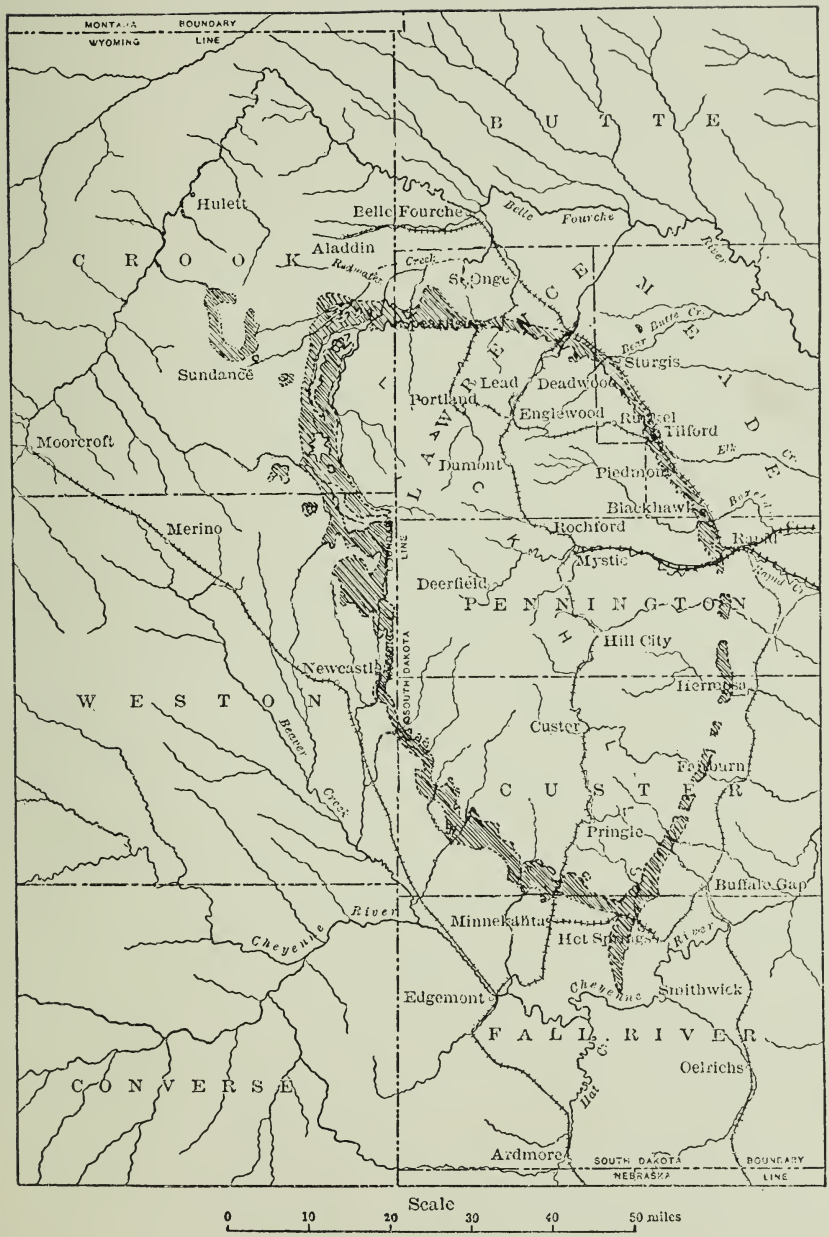
The Black Hills region is bountifully supplied with deposits available for the manufacture of good cement. Until recently little attention has been given to the experimental study of these materials and in this bulletin chief attention is directed toward the examinations of two or three formations of special promise favorably situated with reference to railroads.

The formations chiefly studied are what in geologic literature are known as the Minnekahta limestone, the Graneros shale, and the Niobrara argillaceous chalk. Experimental work indicated that by a proper mixture of materials from the Minnekahta and the Graneros formations a compound can be made, suited in every way for the manufacture of a high grade Portland cement, while the Niobrara can furnish material of proper composition for the making of excellent natural cement. Of the limestones, the Minnekahta is the only one of the region deemed worthy of consideration at this time. The Pahasapa is the great limestone formation of the Black Hills, making long lines of high white escarpment facing on all sides the central part of the uplift, but it contains much maganesia and too little attention has been paid to the possible magnesia-free portions to allow for any reliable statement as to favorable localities where this limestone might be profitably exploited. Such other limestones as occur within the region are thin or impure and are often inclined to considerable

variation in character, hence have not been given special attention.

The shale-bearing formations are abundantly represented and several of them give evidence of possible value used in combination with the Minnekahta limestone. In addition to the Graneros already mentioned there are the Pierre, the Niobrara, the Carlile, the Morrison, and possibly other formations that merit careful examination, should an exhaustive study be made of all available materials. That the geologic position of the various formations may be better understood a complete generalized geologic section of the Black Hills region is here given.

The Minnekahta Limestone. The Minnekahta limestone, known commonly in early geological papers as the Purple Limestone, although seldom more than fifty feet in thickness is one of the prominent outcropping formations of the Black Hills region. It is a close-textured, thinly-bedded, grayish limestone with frequently a slight pinkish or more often a purplish tinge, hence the earlier name. The bedded nature of the rock is much concealed on fresh surfaces, but weathering reveals the condition in a very definite way. Near the middle of the formation the nature of the bedding is such as to allow for slightly more rapid weathering than elsewhere and this feature generally reveals itself in unmistakable manner. Near the bottom the bedded nature is again pronounced and the rock here contains considerable argillaceous material. The lowest beds which are generally much ironstained are not commonly exposed. In some localities where they are exposed the limestone breaks up into numerous small lenticular concretionary bodies within which fairly well-preserved fossils are found. In general, however, the formation is markedly free from fossils, although not quite so much so as was earlier supposed. Mr. N. H. Darton of the United States Geological Survey records the identification of *Bakewellia, Edmondia,* and *Nuculana.*



Map showing outcrop of the Minnekahta formation

The Minnekahta is underlain by a thin series of soft clay shales known as the Opeche formation and is overlain by a thicker series of soft sandy shales, the Spearfish formation, commonly known as the Red Beds. The dip, sometimes steep, sometimes low, varies in a more or less undulatory manner, but in general it follows the nature of all of the periclinal sedimentary rocks of the region in that it is distinctly apocentral, that is, it dips outwardly on all sides from the higher central portion of the main Balck Mills uplift.

The outcrops are unusually prominent every where. The relatively hard, more or less sharply dipping limestone between the soft shale beds readily lends itself to the production of a bold topography. Hundreds of streams, constant and intermittent flowing radially from the central area of the uplift run across the formation and each carves for itself a V-shaped gateway to the red valley beyond. On the outer slopes wherever the dip is considerable, the rock is nearly bare of soil and vegetation although the pine trees, Pinus ponderosa, cling tenaciously to it wherever a roothold can be secured. The inner line of outcrops is a continuous succession of prominent escarpments which, facing the central area of the uplift dome, stand out in bold relief as they overlook the sharply, trenched Opeche below.

The width of outcrop varies greatly. Where the dip is steep as for example along Beaver creek and the lower portion of Gillette canyon east of Newcastle and on the immediate flanks of several of the lacolithic areas which interrupt the general foothill structure, the width of the outcrop is reduced to a minimum. Elsewhere where the dip is slight and gently undulating, such as in the southern Hills west and northwest of Hot Springs, in the northern Hills near Spearfish and still better, along the western side of Cold Spring creek in Wyoming extensive areas are capped by this limestone. Local irregular and isolated

FORMATION	CHARACTER	AVERAGE THICKNESS FEET	AGE
Laramie	Massive Sandstone and Shale	2,500	Tertiary and Pleistocene
Fox Hills	Sandstone and Shale	250-500	Cretaceous do
Pierre Shale	Dark Gray Shale	1,200	do
Niobrara	Chalk and Calcareous Shale	225	do
Benton Group—			
Carlile formation	Gray Shales with thin sandstones, limestone and concretionary layers.	500-750	do
Greenhorn Limestone	Impure, slabby limestone	50	do
Graneros Shale	Dark shale with lenses of massive sandstone in its lower part at some places	900	do
Dakota Sandstone	Massive buff sandstone	32-150	do
Fuson	Very fine grained sandstone and massive shale. White to purple color	30-100	do
Minnewasta Limestone	Gray limestone	0-30	do
Lakota	Massive buff sandstone with some shale	200-350	do
Morrison Shale	Pale, grayish green shale.	0-150	Jurassic
Unkpapa Sandstone	Massive sandstone white, purple, red buff	0-250	do
Sundance	Thin limestones. Dark drab shales and buff sandstones. Massive sandstone at base	60-400	do
Spearfish	Red, sandy shale with gypsum beds	350-500	Triassic
Minnekahta Limestone	Thin-bedded, gray or purple limestone	30-50	Permian
Opeche	Red, slabby limestone and sandy shale	90-130	Permian
Minnelusa	Sandstones, mainly buff and red; in greater part calcareous. Some shales and thin limestones included	400-450	Carboniferous
Pahasapa Limestone	Massive gray limestone	250-500	do
Englewood Limestone	Pink, slabby limestone	25	do
Whitewood Limestone	Buff colored, mottled limestone, withgreenish shales at top	0-80	Ordovician
Deadwood	Red-brown quartzite, calcareous shales and sandstones, locally conglomeratic, partly massive	4-150	Cambrian

areas are common. Such are the tongue-like extension between Hot Springs and Alabaugh canyon, the elongated irregular dome alongside the Chicago & Northwestern railway northwest of Rapid City, and the more or less circular outcrop about Green mountain, Strawberry mountain, Inyan Kara, Warren peaks, Elkhorn peak, Crook mountain Bear Butte, etc.

The formation affords many fine springs, a feature of much interest. It is the source of the abundant thermal waters at Hot Springs and Cascade springs and of colder waters at many other places. Analyses as elsewhere given show this limestone to be of excellent character for various industrial uses. It serves for the manufacture of high grade lime and is indeed the chief source of such material for the Black Hills region. It was used for a considerable time in the smelter at Rapid City in the reduction of the siliceous gold ores of the northern Hills, the limestone being obtained alongside the Chicago & Northwestern railroad three miles northwest of Rapid City. It is now being extensively utilized in the cyanide plants of the Homestake Gold Mining Company in Lead, their two kilns of modern make being constantly in operation at the quarry on Elk creek near Doyle on the Burlington railroad a short distance above Piedmont. The following analyses of this limestone indicate its superior nature in the preparation of a high-grade Portland cement. Analysis No. 1 is by Professor M. F. Coolbaugh of the School of Mines, the material coming from an outcrop alongside the Missouri river and Northwestern railroad near the old Canyon Lake site, four miles west of Rapid City. Analysis No. 2 and No. 3 are from samples taken near Newcastle. These two analyses are given in Bulletin No. 315 of the U. S. Geological Survey.*

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*Ball. Sydney H. Portland Cement Materials in Eastern Wyoming. Bulletin No. 315, U. S. Geol. Survey, p. 232-244.

Table No. 2.

Analysis No. 1.

Silica, SiO_2	1.21 Percent
Ferric Oxide, Fe_2O_3	0.16
Alumina, Al_2O_3	0.47
Titanium oxide, TiO_2	trace
Lime, CaO	54.56
Magnesia, MgO	0.41
Sulphur, S	0.11
Alkalies, K_2O,	trace
Na_2O,	trace
Phosphorus pentoxide, P_2O_5	trace
Carbon dioxoxide, CO_2	42.30
Water, H_2O	0.91
Total	100.13

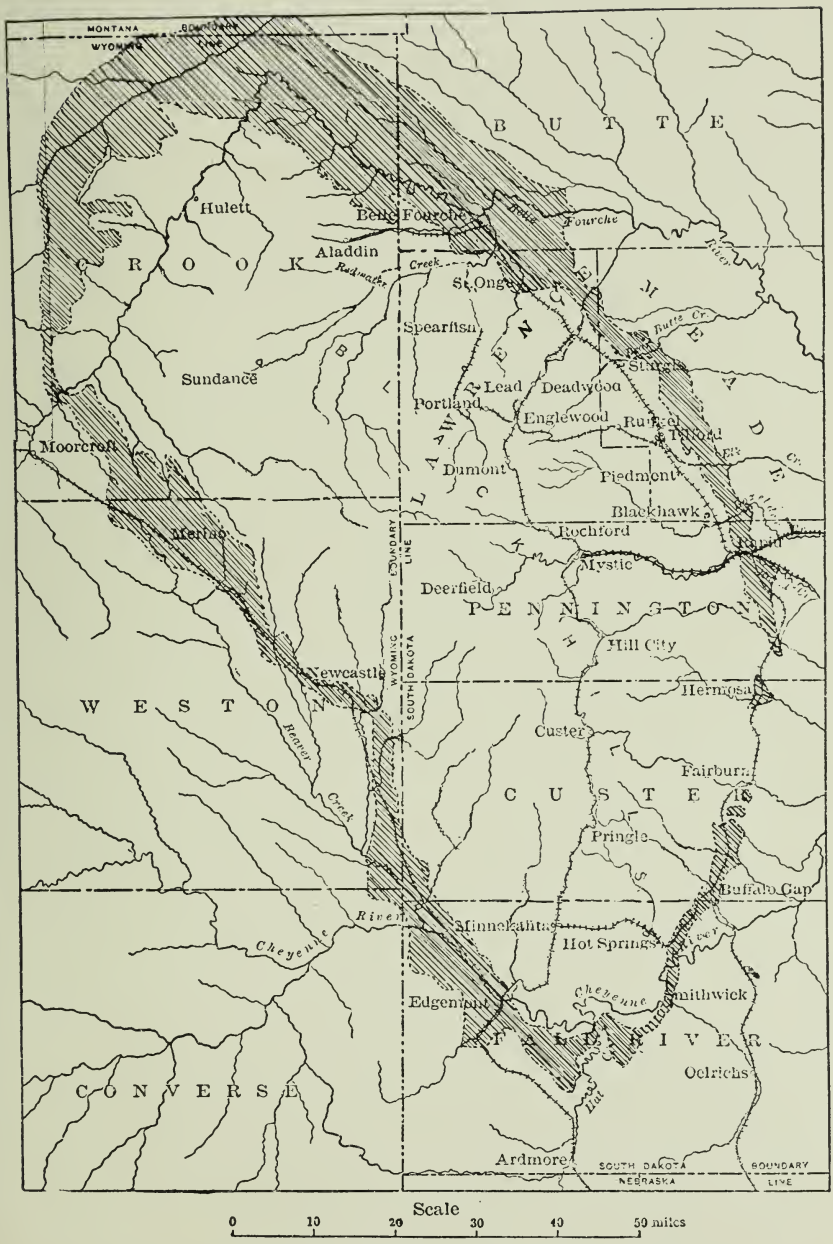
Loss on ignition 42.95 percent.

Table No. 2 A.

Analysis Nos. 2 and 3.

	No. 1.	No. 2.
Silica, SiO_2	1.08 Percent	1.42 Percent
Alumina, Al_2O_3	0.33	0.68
Ferric oxide, Fer_2O_3	0.77	0.40
Manganese oxide, MnO,	0.46	0.11
Lime, CaO,	53.40	52.85
Magnesia, MgO,	0.57	0.72
Sulphuric anhydride, SO_3,	0.12	0.12
Alkalies, Na_2O,	0.36	0.76
K_2O,	0.16	0.30
Water at 100° C	0.20	0.10
Ignition loss	42.92	42.78
Total	100.37	100.24

The Graneros Shale. The Graneros shale is the lowest member of what is frequently known as the Benton group. Lying as it does just outside the main Cretaceous hogback its outcrop completely encircles the Black Hills uplift as a broad belt of low undulating hills and valleys and serves as the innermost fringe of the great plains of the Black Hills region.



Map showing outcrop of the Graneros formation

The formation is made up distinctively of fine-grained dark gray to black shale, but there is considerable modification of this simple character in many places, especially in that portion of the area lying within the borders of Wyoming. Here a well-defined four-fold character generally shows itself, viz., first, a lower portion of fine black fissile shale 150 to 300 ft. thick; above this, massive grayish-buff sandstone of varying thickness up to 40 ft.; following this a series 250 to 300 ft. thick of dark shales of hard texture which weather to a distinct grayish white color; lastly, at the top a series of very black shales 400 to 500 feet thick, much resembling the shales of the lowest member. In that portion of the Black Hills region lying within South Dakota the four fold character is less prominent and particularly is this true so far as concerns the eastern and southern portions of the uplift.

The sandstone member varies greatly in thickness and hardness; sometimes it stands out in bold cliffs much like the Dakota sandstone whose texture and grayish-buff color it closely simulates. Elsewhere it is much concealed and over considerable areas it is thin or absent. Near Newcastle where this member reaches local prominence the rock contains petroleum as indicated by certain bore holes and by small flows from several natural springs. The sandstone shows in various places along the eastern side of the Black Hills, as, for example, at Rapid City and Hermosa, but here it partakes more of the nature of greatly elongated lenses and, except for a few lenticular hills, is of little or no topographic significance.

The hard grayish shale member, now commonly designated as the Mowrie beds, from Mowrie creek near Buffalo, Wyoming, where it was first studied, is prominent in much the same areas as the underlying sandstone except that it partakes less of the lenticular nature of the latter. Its greatest thickness is apparently in the vicinity of Belle Fourche and northwest of there along the Belle Fourche and the Little Missouri

rivers. Ridges often result from the more rapid wearing away of the softer shales and these ridges by contrast of color, by topographic importance and by their general wooded covering serve admirably in delineating the outcrops of these beds from the generally non-wooded darker shales of the lower and the upper members. The beds are further characterized by the presence of multitudes of fish scales in almost all good exposures. Southeast of Belle Fourche the gray color and the indurated nature become gradually less distinctive and the fish scales grow fewer in number until at Rapid City only the closest scrutiny establishes the presence of any portion of the member.

The lowest member needs little more character description than already given. With the exception of the passage beds of alternating shales and sandstones near the bottom and occasional bands of fair to good sized lime-clay concretions it is practically entirely a fine fissile, black shale. It lends itself to especial attention here in that the shale material used with limestone from the Minnekahta formation in the preparation of the Portland cement described elsewhere in this bulletin was obtained from this member. The following chemical analyses shows its favorable character. No. 1 is by Professor Coolbaugh of the Shool of Mines and represents material obtained a few hundred yards north of the Hinrichs-Lanphere saw mill in Rapid City. No. 2 represents material from near Newcastle and is recorded in Bulletin No. 315 of the U. S. Geological Survey.*

*Ball. Sydney H. Portland Cement Materials in Eastern Wyoming. Bulletin No. 315, U. S. Geol. Survey, p. 232-244.

Table No. 3.

Analysis No. 1

Silica, SiO_2,	58.74 Percent
Ferric oxide, Fe_2O_3	3.87
Alumina, Al_2O_3,	18.97
Titanium oxide, TiO_2,	0.71
Lime, CaO,	0.93
Magnesia, MgO,	1.62
Sulphur, S,	0.21
Alkalies, K_2O,	1.49
Na_2O,	0.58
Phospheros pentoxide, P_2O_5	1.44
Manganese oxide, MnO,	trace
Loss on ignition	11.93
Total	100.24

Table No. 4.

Analysis No. 2.

Silica, SiO_2	58.82 Percent
Alumina, Al_2O_3,	16.43
Ferric oxide, Ee_2O_3	4.47
Manganese oxide. MnO.	0.24
Lime, CaO,	0.54
Magnesia, MgO,	1.68
Sulphuric anhydride, SO_3	1.32
Alkalies, Na_2O,	0.33
K_2O,	2.18
Water at 100° C,	6.39
Ignition loss,	7.93
Total	100.33

There is little or no reason for believing that much of the shales of the higher members of the formation would not lend themselves as freely to the production of good cement as the lowest member, although little experimental work has been carried on by way of demonstrating their real value for such purpose.

The upper black shale member much resembles the lowest member. It is generally thicker and is inclined to be more abundantly supplied with lime-clay

concretions. These concretions sometimes reach a diameter of five or six feet or more. Thin bands of limestone are occasionally found. Some of these are highly fossiliferous and, while never of great thickness, they occasionally are able to control the topography sufficiently to outline ridges and hills of some prominence.

The formation taken as a whole is well exposed. The outcrop is generally wide. This is more particulary true around the northern end and along the western side of the Hills where for many miles it serves as a wide valley bed for the Belle Fourche and the Little Missouri rivers and their tributaries, the width of outcrop in places reaching ten or a dozen miles or more. Along the eastern foothills the outcrop is narrower. From Whitewood creek southward to Spring creek it varies from one to four or five miles. South of Spring creek it is much concealed by unconformable Tertiary deposits. Throughout the entire region alluvial deposits cover up considerable areas along the streams and higher gravels veneer many of the gentle slopes and flat topped hills and ridges. The alluvial deposit is particularly prominent along the Belle Fourche river and the Little Missouri river in the northern and northwestern part of the area and along lower Beaver creek and certain portions of the Cheyenne river in the southern part. This covering prevails to a less extent elsewhere but is of significance wherever the formation is crossed by streams of a meandering nature.

The Graneros, like the Minnekahta, is favorably situated with reference to railroads. Northwest of Newcastle and between Newcastle and Edgemont the Burlington is built upon this formation, for many miles. This road could easily reach it by short extension near Piedmont or near Spearfish if such should be desired. At Rapid City the formation is convenient to the Missouri river and Northwestern, to the Chicago, Milwaukee and St. Paul, and to the two lines of

the Chicago & Northwestern. South of Rapid City to Buffalo Gap the Chicago & Northwestern touches it at many points. North of Rapid City to Whitewood this road lies within the red valley but even here easy access is permitted to the Graneros by the many notches through the intervening hogback ridges. North of Whitewood the Belle Fourche branch of the Chicago & Northwestern railroad quickly passes outside the red valley to the formation. It then follows the general direction of outcrop to and beyond Belle Fourche, being flanked on both sides by exposures in most convenient manner. In much the same way west of Belle Fourche the formation lies convenient to the Wyoming and Missouri River railroad.

The Niobrara Formation. The Niobrara formation lies just beneath the Pierre shale. Compared to the Pierre it is thin, being seldom more than 225 ft. thick and around the northern end of the uplift it is approximately 100 ft.

The material is very soft, is highly calcareous, and partakes more or less of the nature of an easily pulverized chalky limestone. Fresh material is of a bluish-gray color but outcrops are generally well weathered and disintegrated and in this condition the material is of a rich creamy yellow color.

The formation seldom develops any topographic features of significance but its pronounced yellow color renders the formation conspicious whenever the outcropping surface is broken. The fossil oyster, *Ostrea congesta,* occurs in abundance in occasional thin indurated bands and this also serves as a distinctive feature.

Near the head of the Little Missouri river west of the Bear Lodge range and in the vicinity of Newcastle where local disturbances have given the formation a steep dip the width is only a few yards but frequently elsewhere it reaches one mile or more.

In view of the fact that the formation outcrops continuously and in a fairly regular manner around

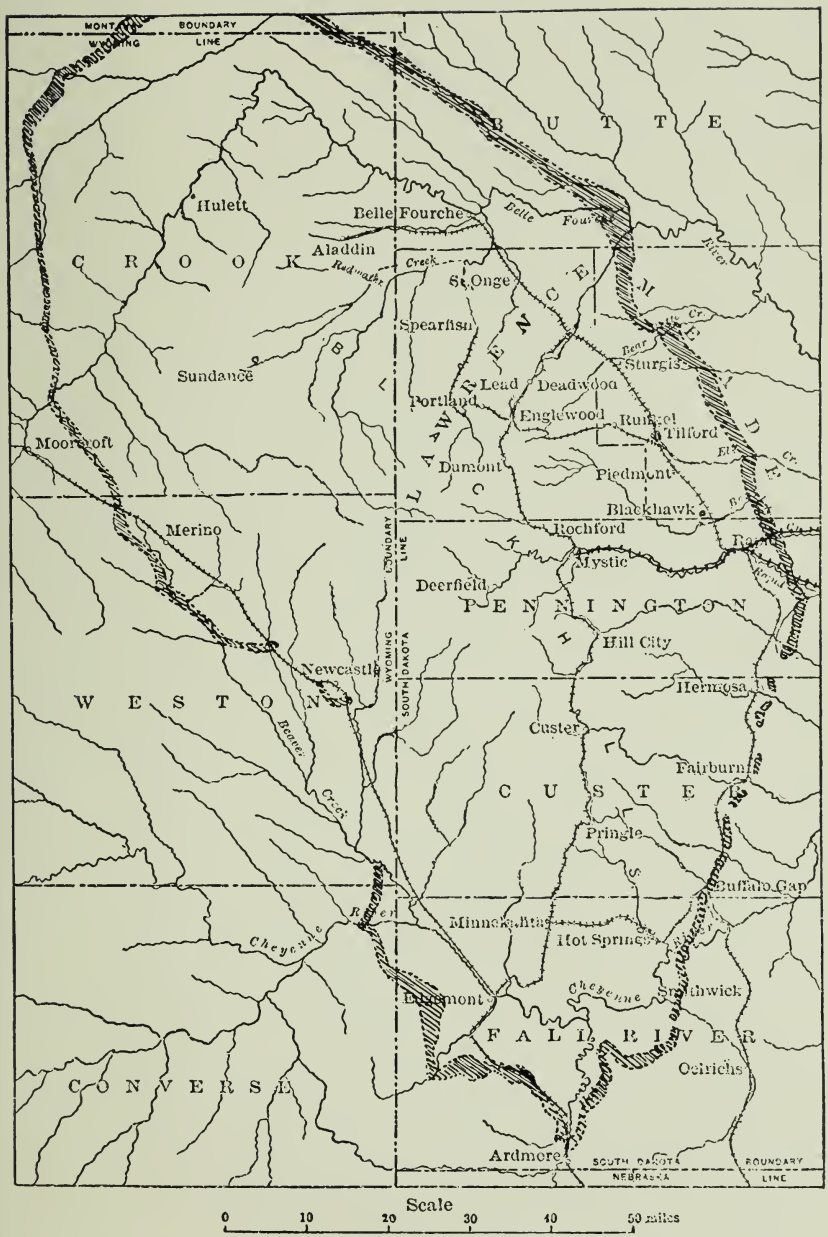
the Black Hills and is so readily distinguished in the field it serves nicely as a stratigraphic guide and delineates in an excellent manner the plainward extent of the more pronounced structural features of the Black Hills uplift.

The Niobrara is the formation that affords much of the raw material for the Portland cement plant at Yankton in the southeastern part of the state. The interesting feature with reference to the Black Hills area is that the chemical nature of the rock as revealed by analysis, while not uniform, indicates that this formation can furnish in places within the Black Hills area a most excellent material for natural cement. Its physical condition is almost everywhere particularly favorable. The shaly pulverent nature of the material would allow for easy excavation and doubtless steam shovel operation could be carried on to considerable depth. The chemical nature varies and is not always satisfactory but the following analysis by Professor Coolbaugh of the School of Mines from material near Antelope creek ten miles east of Tilford shows a particularly favorable composition and indicates the possibility of utilizing this material to most excellent advantage:

Table No. 5.

Silica, SiO_2,	15.51 Percent
Ferric oxide, Fe_2O_3 and Alumina, Al_2O_3	5.80
Lime, CaO,	38.85
Sulphur, S	trace
Magnesia, MgO,	1.08
Alkalies, K_2O, and Na_2O,	1.50
Loss on ignition	36.67
Total	99.41

This may be more readily compared with the analysis of Portland cement manufactured from the Minnekahta-Graneros mixture given elsewhere in this bul-



Map showing outcrop of the Biobrara formation

letin by recalculating so as to omit reference to loss on ignition. The analysis thus arranged is as follows:

Table No. 6.

Silica, SiO_2,	24.65 Percent
Ferric oxide, Fe_2O_3 and Alumina, Al_2O_3	9.16
Lime, CaO,	61.36
Sulphur, S, (estimated)	0.20
Magnesia, MgO,	1.72
Alkalies, K_2O and Na_2O,	2.52
Total	99.61

Table No. 7.

	No. 4. Near Rapid City.	No. 5. Bear Butte Piere
Silica SiO_2,	22.47	6.81
Ferrie Oxide and Alumina	9.80	3.23
Lime CaO,	25.01	46.75
Magnesia, MgO,	1.54	0.93
Alkalies,	0.75	1.43
Sulphur,	1.84	trace
Loss on ignition	38.25	39.56
Total	99.66	99.71

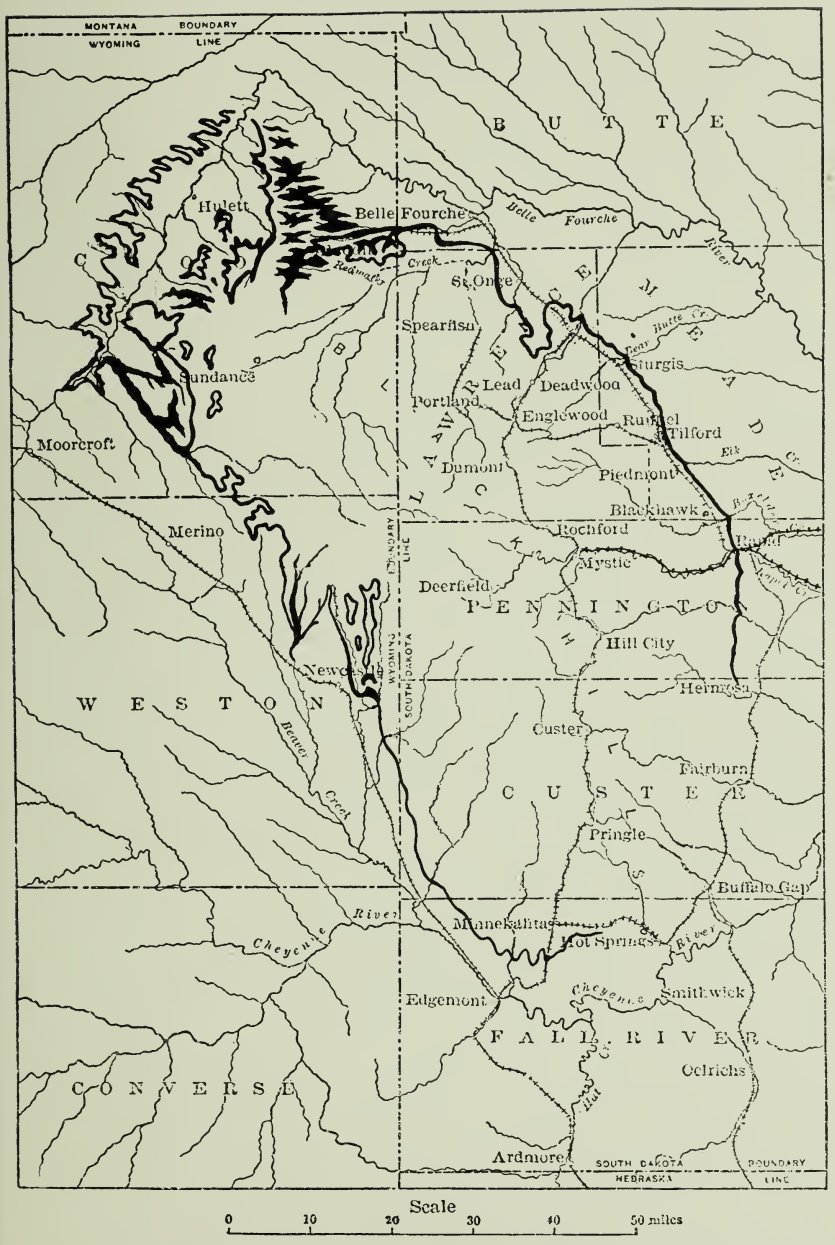
The Burlington and Missouri River railroad follows the outcrop of the formation for some miles near Ardmore and near Newcastle and crosses it again near Moorcroft. The Chicago & Northwestern railroad crosses it near Buffalo Gap, then follows it fairly closely to Rapid City. Just east of Rapid City the Pierre, Rapid City & Northwestern cuts across the formation in a particularly favorably manner while the Chicago, Milwaukee & St. Paul could reach similarly good outcrops near by with little side trackage. Between Rapid City and Whitewood the formation outcrops in a wide belt several miles east of the railroad but this could be easily reached if desired through the various gaps in the hogback ridges.

The Morrison Formation. The Morrison formation of early Cretaceous or late Jurassic age lies just beneath the Lakota sandstone. It does not completely encircle the Hills, there being a considerable space in the southeastern portion of the uplift where it is absent.. Along the eastern side of the Hills it is under lain by the massive soft Unkapapa sandstone but along much of the western portion of the uplift the Unkpapa is absent and it is underlain by the Sundance formation.

The formation is made up of shales and thin nodular bands of white argillaceous limestone, the shales greatly predominating. Sometimes the clay-lime nodules are of little or no significance and occasionally thin shaly sandstones appear, the latter showing best in the Bear Lodge mountains. The shales carry more or less carbonaceous matter and vary much in color. Olivine green predominates in fresh exposures but pale greenish gray, yellowish gray, pink, red, maroon, chocolate, purple, and black are not infrequently observed. A characteristic and fairly common feature of the formation is the presence of large saurian bones near the bottom of the formation. Several localities have afforded good specimens.

The formation where best exposed varies in thickness up to 160 feet or more. In the Bear Lodge mountains and southward it varies from 40 ft. to 160 ft. Along the northeastern side of the Hills the thickness varies in much the same way. On the Oelrichs quadrangle it is absent.

Exposures are good in many places and convenient to the railroads. The Chicago & Northwestern parallels the outcrop all the way from Rapid City to Whitewood and could easily tap many of the exposures by short spur trackage, the outcrops being all to the east of the track and at various convenient heights above the main line. The Burlington and Missouri River railroad crosses the formation between Edge-



Map showing outcrop of the Morrison formation

mont and Minnekahta and is fairly convenient to it near Newcastle and Piedmont. The Wyoming and Missouri River railroad follows the formation for some distance near Aladdin.

Little experimental study has been given this formation but it is worthy of consideration. An analysis* of material obtained near Newcastle gives the following favorable composition:

Table No. 8.

Silica, $Si O_2$,	45.78 Percent
Alumina, $Al_2 O_3$	12.92
Ferric oxide, $Fe_2 O_3$	3.95
Manganese oxide, MnO,	0.33
Lime, CaO,	0.56
Magnesia, MgO,	0.73
Sulphuric anhydride, SO_3	0.48
Alkalies, $Na_2 O$,	0 64
$K_2 O$,	0.50
Water at 100° C,	8.26
Ignition loss, largely carbonaceous matter	26.32
Total	100.42

The Carlile Formation. The Carlile formation outcrops immediately within the Niobrara and constitutes the upper portion of the Benton group. It is made up of a series of thin black shales with occasional thin impure sandstone and limestone bands. In places the limestone is quite argillaceous and takes on the form of large concretionary or lenticular masses. These sometimes contain fossils.

In the Devils Tower quadrangle the formation is made up of three fairly distinct divisions, viz., an upper division, chiefly shale, 300 feet thick; a middle division 125 feet thick, concretions and shale; and a lower division 200 feet thick, mostly shale. Elsewhere this threefold nature is absent or is not sufficiently prominent to disclose itself in any characteristic man-

*Ball. Sydney H. Portland Cement Materials in Eastern Wyoming. Bulletin No. 315, U. S. Geol Survey, p. 236.

ner. The total thickness varies from about 400 feet near Buffalo Gap to about 700 feet in the vicinity of Newcastle. The shale portions much resembles the shales of the Pierre and the Graneros and doubtless the chemical composition is much the same.

The formation is readily accessible to the various railroads in much the same manner as the overlying Niobrara. Since it has not been carefully studied as to possibilities in the way of affording ingredients for cement manufacture further description need not be given at this time.

The Pierre Formation. The Pierre formation is the latest Cretaceous formation that outcrops within the immediate Black Hills region. It completely encircles the uplift as a broad belt and is one of the greatest formations of the Plains region. West of the Hills its outcrop averages perhaps five or six miles in width. Northeast, southeast, and south of the Hills it reaches several times this width while to the east of the Hills it extends far beyond the limits of the regions under discussion. Every railroad entering the Hills traverses the formation for several or many miles and good exposures are abundant and convenient.

The thickness of the formation is given as approximately 1200 feet. The material is almost wholly black shale. Weathered surfaces show lighter color and in the lower portion where some lime enters into the composition the color is inclined to a dull yellowish color. Lime and lime-iron concretions are abundant at several horizons. These are often highly fossiliferous. particularly those in the upper part of the formation and the invertebrate forms that they contain are varied in character and often beautifully preserved. The formation is abundantly exposed and readily distinguished. For further details of structure, distribution and phsical character of this formation as well as for the Carlile the reader is referred to the various publications listed on a subsequent page.

Little attention has been given to the accurate chemical nature of the shales. One analysis* made from material obtained one mile above Spencer siding on the Burlington and Missouri River railroad southly known as the "red beds." This formation completesoutheast of Newcastle, is as follows:

Table No. 9.

Silica, SiO_2,	60.66 Percent
Alumina, Al_2O_3	22.13
Ferric oxide, Fe_2O_3	1.21
Manganese oxide, MnO,	0.44
Lime, CaO,	1.59
Magnesia, MgO,	1.54
Sulphuric anhydride, SO_3	0.43
Alkalies, Na_2O,	0.53
K_2O,	3.16
Ignition loss	9.28
Total	99.97

The above analysis shows certain undesirable percentage of ingredients but field observation furnish good reason for believing that a series of analyses of material selected from various localities would show a fair number with favorable proportions of the various important elements.

Gypsum. A brief word concerning the nature and occurrence of Black Hills gypsum may be of interest here in view of the fact that gypsum enters in a minor way into the manufacture of cement.

The deposits of Black Hills gypsum occur at various horizons within the Spearfish formation, popularly known as the "red beds." This formation completely encircles the Hills and good exposures seldoms fail to show gypsum, nearly pure white, in sufficient quantity and of sufficient purity to be of commercial value. The deposits are extensive and convenient. They are sometimes lenticular but more often they occur in beds

*Ball. S. H. Portland Cement Mat. in Eastern Wyoming. Bul. 315, U .S. G. S. p. 238.

of nearly uniform thickness and in some places where the outcrop is favorable these white beds may be traced across the landscape for many miles as they stand out in prominent contrast to the enclosing red sandy shales.

The gypsum is of a high degree of purity and for several years has been utilized in the manufacture of stucco or plaster. Two plants are now in active operation, one at Hot Springs and one near Rapid City. The Hot Springs plant has been in interrupted operation for several years. The Rapid City plant was placed in commission early in the year 1908. Both plants are convenient to large bodies of good raw material.

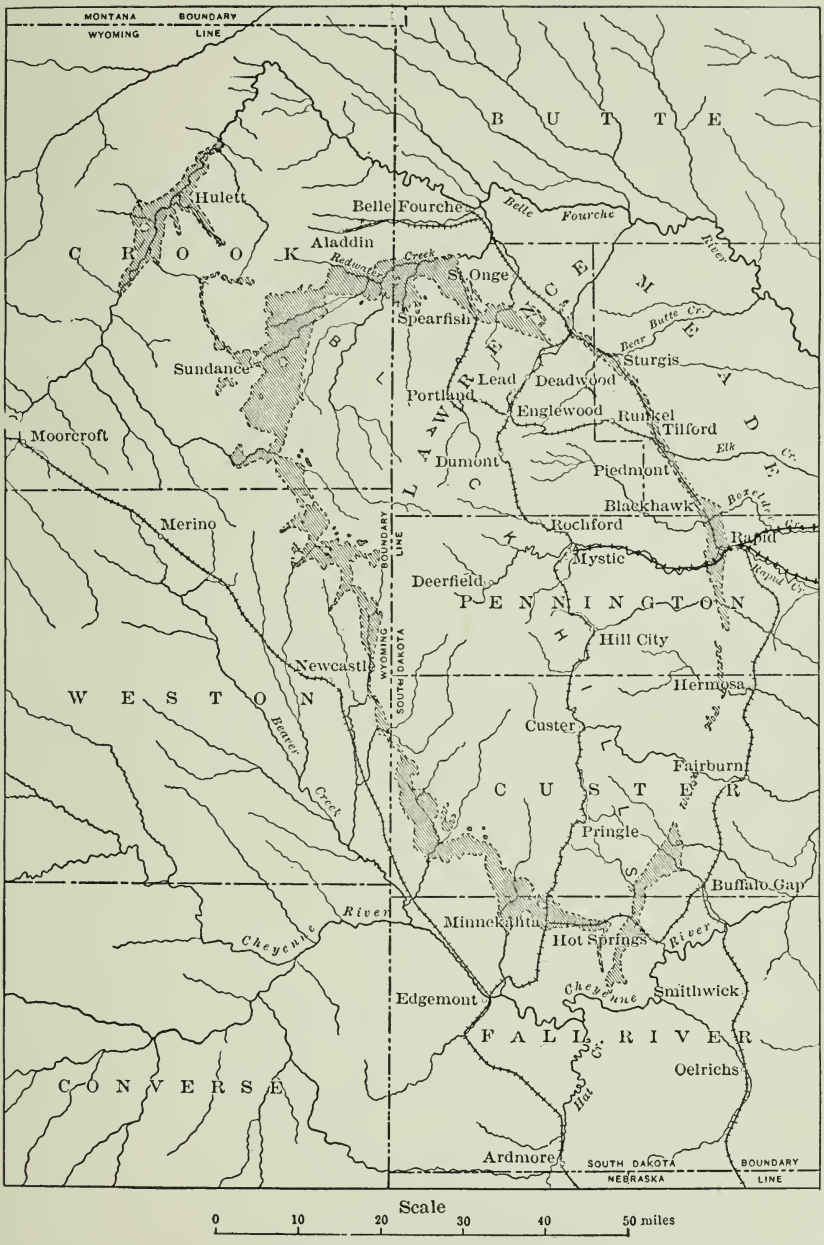
Analysis of the Hot Springs gypsum made by Professor Coolbaugh show the following composition. No 1 is from a thirteen foot bed near the bottom of the exposure and No. 2 is from a four foot, the two being separated by one foot of red shale carrying only thin seams of gypsum. Both beds are utilized.

Table No. 10.

Silica, SiO_2,	0.12	0.09 Percent
Ferric oxide, Fe_2O_3 Alumina, Al_2O_3	0.12	0.06
Sulphuric anhydride, SO_3	47.77	44.86
Lime, CaO,	33.00	32.89
Magnesia, MgO,	0.10	0.28
Loss on ignition	20.85	21.41
Totol	101.97	98.59

Mr. Steiger of the U. S. Geological Survey laboratory gives the following analysis of material from near Cascade Springs:*

*Darton, N. H. Gypsum Deposits in South Dakota. Bulletin No. 223, U. S. Geological Survey, p. 76-78.



Map showing outcrop of the gypsum-bearing Spearfish formation

Reproduced from U. S. G. S. Bulletin No. 223. New railroads inserted

Table No. 11.

Lime, CaO,	32.44 Percent
Magnesia, MgO,	0.33
Alumina, Al_2O_3	0.12
Silica, SiO_2	0.10
Sulphuric anhydride, SO_3	45.45
Carbon dioxide, CO_2,	.85
Water, H_2O,	20.80
Total	100.09

Additional Literature. The reader who may wish to examine more fully into the details of distribution and description of the various geological formations described or mentioned in this paper will find much additional information in the following atlas folio publications of the U. S. Geological Survey:

Darton, N. H., Oelrichs folio. No. 85, 1902.

Darton, N. H. Newcastle folio. No. 107, 1904.

Darton, N. H., and Smith, U. S. T. Edgemont folio No. 108, 1904.

Darton, N. H., Sundance folio, No. 127, 1905.

Darton, N. H. and O'Harra, C. C. Aladdin folio, No. 128, 1905.

Darton, N. H., and O'Harra, C. C., Devils Tower folio No. 150, 1907.

PART II.

OUTLINE FOR THE ANALYSIS AF CEMENTS AND CEMENT MATERIALS.

M. F. COOLBAUGH, M. A.

Outline for Analysis of Cement and Cement Materials.

M. F. COOLBAUGH, M. A.

The outline scheme for the analysis of cements will serve in many places as a scheme for the analysis of limestones, raw mixtures, shales and clays.

Decomposition.—With highly silicious material decomposition can be effected by treatment with hydrochloric and nitric acids, filtration, fusion of the residue with sodium carbonate, and solution of the fused mass in water and hydrochloric acid. Or the fusion may be made directly without a previous treatment with acids. For the decomposition of limestones or unburned mixtures, ignite material with one-half to an equal weight of sodium carbonate and treat mass with diluted hydrochloric acid.

Silica.—Two evaporations are essential for this determination when considerable accuracy is required. Even then a complete separation is not effected, as some silica will be found with the iron and alumina precipitate. Some alumina will also be found with the dehydrated silica. Corrections for these small amounts of impurities are not, however, necessary in technical work, since the weights tend to counterbalance each other. For greater accuracy, the silica should be driven off by treatment with hydrofluoric acid and a few drops of sulphuric acid. The residue should be

added to the precipitates from the ammonia treatment.

Iron and Alumina.—The precipitates of iron and aluminum hydroxides, which contain also titantic-acid, ferric phosphate, and a small amount of silica, is purified from calcium and traces of manganese by solution in nitric acid and reprecipitation with ammonia. The calcium in the first precipitate is caused by impurities of ammonium carbonate in the ammonia, or by carbon dioxide in the air of the laboratory. The manganese is not separated even by two precipitations unless the content of that element is small. If this is not the case, separate it from the iron and aluminum by precipitation of the latter as basic acetates in a dilute acetic acid solution containing sodium acetate. The amount of silica is separated after the fusion with the acid sodium sulphate, as in (3), and evaporation of the sulphuric acid solution to low bulk. The weight found here should be added to that already found.

Titanium Oxide.—The titanium is determined in the solution from the titsation of iron in (3). Add ten c.c. hydrogen peroxide, make to a definite bulk, and compare color with that of a standard solution of titanium treated in a similar manner.

Manganese.—This is determined in the filtrate from the iron and alumina precipitation. It is precipitated with ammonium sulphide, dissolved in nitric acid and determined colorimetrically by use of silver nitrate and ammonium persulphate.

Lime.—Acidify with hylrochloric acid the combined filtrates from the aluminum and ferric hydroxides precipitates, or the filtrate from the manganese determination, heat to boiling, add 10 to 12 c. c. of a saturated solution of ammonium oxalate and slowly neutralize with ammonia. The longer the time taken for neutralization the less danger there is of contamination by magnesium. When greater accuracy is desired, the calcium oxalate is filtered, ignited, dissolved

Scheme for Analysis of Cement.

PART I.

Take 1gr. of the finely ground material and treat with dilute HCl and filter. Dry the residue, fuse with $Na_2(CO_3,)$ take up in water and HCl and add to the main solution. Evaporate to dryness, dehydrate at 120°C, take up in water and HCl and filter. Repeat evaporation and dehydration.

(1) Residue Dry, ignite and weigh as SiO_2

(2) Solution. Make alkaline with NH_4OH, boil and filter. Dissolve percipitate in dilute HNO_3 and reprecipitate with NH_4OH and filter; combine filtrates.

(3) Precipitate $Fe(OH)_3$, $Al(OH)_3$. Dry, ignite and weigh as Fe_2O_3 and Al_2O_3 fuse with $NaHSO_4$, take up in H_2SO_4 reduce with H_2S drive off H_2S with CO_2 and titrate the iron with $KMnO_4$ The iron calculated to Fe_2O_3 and subtracted fron the the combined weights of Fe_2O_3 and Al_2O_3 gives the weight of Al_2O_3.

(4) Solution. Make acid with HCl, heat to boiling, add $(NH_4)_2C_2O_4$, neutralize slowly with NH_4OH and filter.

(5) Precipitate CaC_2O_4. Dry. ignite and weigh as CaO.

(6) Solution. Acidify with HCl, concentrate to 150cc. add 30cc of a saturated solution of $NaNH_4HPO_4$ boil and cool. Then add NH_4OH drop by drop until the precipitate begins to form, and finally add it in slight excess. Allow to stand several hours, filter, wash, ignite and weigh as $Mg_2P_2O_7$

PART II.

Heat 1gr, of the material with one half gr. of Na_2CO_3. Extract with water and filter. Reject residue unless P_2O_5 is to be determined. Acidify solution with HCl, add $BaCl_2$, boil, filter, dry, ignite and weigh as $BaSO_4$,

PART III.

Heat one half gr. of Cement with 4grs. of $CaCO_3$ and one half gr. of NH_4Cl. Extract with water and filter. Reject residue. To the solution add a few drops of $BaCl_2$ and an excess of $(NH_4)_2CO_3$ Filter and reject the precipitate. Evaporate the solution to dryness and drive off the NH_4OH salts. Take up in a small amount of water, add a few drops of $(NH_4)_2C_2O_4$ and filter into a weighed crucible. Evaporate to dryness, add a few drops of HCl, ignite gently and weigh the NaCl and KCl Dissolve in a small amount of water add H_2PtCl_6, evaporate to low bulk, add 85 per cent alcohol and filter. Reject filtrate. Dissolve precipitate in a small amount of hot, water filter into a weighed crucible, evaporate to dryness, dry at 105°C and weigh as K_2PtCl_6 Calculate K_2PtCl_6 to KCl and subtract from the combined weights of the Chlorides to obtain the NaCl.

in hydrochloric acid, made alkaline with ammonia, boiled and the small amount of aluminum hydroxide filtered out and added to that already found. The calcium is reprecipitated as the oxalate, filtered, ignited and weighed as calcium oxide, or filtered, washed, dissolved in sulphuric acid and the oxalic acid titrated with potassium permanganate.

Magnesia.—For an accurate determination of this substance, the first precipitate should be dissolved in hydrochloric acid, two c. c. of a saturated solution of sodium ammonium phosphate added, and then ammonia, drop by drop, as described in (6) in the scheme. The reason for the second precipitation is that the normal magnesium ammonium phosphate is not the only precipitate formed in the presence of a large excess of ammonium salts, but is contaminated by some $(NH_4)_4\ Mg\ (PO_4)_2$ which yields $Mg_2\ P_2O_7$ only on very strong ignition.

Sulphur.—Ignite the material with one-half its weight of sodium carbonate and a small amount of sodium nitrate, extract with water, filter, acidify filtrate with hydrochloric acid, and precipitate the sulphur, with barium chloride as barium sulphate. The sodium nitrate is added to oxidize the sulphur from the the sulphide to the sulphate condition.

Phosphoric Anhydride.—For this determination treat the residue obtained in the sulphur determination with nitric acid, evaporate to dryness to remove silica, take up in dilute nitric acid, filter, treat residue with hydrofluoric and nitric acids, finally with nitric acid alone and unite solutions. Precipitate the phosphorus with ammonium molybdate, dissolve precipitate in dilute ammonia, acidify with sulphuric acid, reduce with zinc and titrate with potassium permanganate.

Carbon Dioxide.—Decompose the material with hydrochloric acid and absorb the carbon dioxide in soda lime or a solution of potassium hydroxide.

Alkalies.—For this determination follow the well

known method of Professor J. Lawrence Smith, the outline of which is given in the scheme.

Insoluble Residue.—Decompose material slowly with a 10 per cent solution of hydrochloric acid, filter, dry, ignite and weigh the residue.

Loss on Ignition.—Blast a weighed sample for fifteen minutes in a covered platinum crucible. Cool and weigh. Check the loss by blasting for another five minutes.

REFERENCES.

Report of the Sub-Committee on Uniformity in Analysis of Materials for the Portland Cement Industry. J. Soc. Chem. Ind. Vol. 21, p. 12-30.

Analysis of Materials for the Portland Cement Industry. By W. F. Hillebrand. J. Am. Chem. Soc. Vol. 25, p. 1180.

The Analysis of Portland Cement. By Bertram Blount. J. Am. Chem. Soc. Vol. 26, p. 995.

The Technical Analysis of Cements. By S. F. Peckham. J. Am. Chem, Soc. Vol. 26, p. 1636.

The analysis of Silicate and Carbonate Rocks. By W. F. Hillebrand. Bulletin 305, U. S. Geological Survey.

PART III.

THE PREPARATION OF THE CEMENT FROM THE RAW MATERIAL FOR EXPERIMENTAL PURPOSES.

C. H. FULTON, E. M.

The Preparation of the Cement.

C. H. FULTON, E. M.

The raw material, its extent and composition, for the manufacture of the cement has been described in in Part I of this bulletin.

The limestone used in making the cement described below is from a cut on the Missouri River and Northwestern R. R. near Canyon Lake about four miles west of Rapid City and has the following analysis.

Silica	1.21
Ferric oxide	.16
Alumina	.47
Titanium oxide	trace
Lime	54.56
Magnesia	.41
Sulphur	.11
Alkalies	trace
Phosphorous pentoxide	trace
Carbon dioxide	42.30
Water above 100° C.	.91
	100.13
Loss on ignition	42.95

The shales used is from the foot hills, one-half mile northwest from the center of Rapid City and has the following analysis:

Silica	58.47
Ferric oxide	3.89
Alumina	18.97
Titanium oxide	.71
Lime	.93
Magnesia	1.62
Sulphur	.21
Potassium oxide	1.49
Sodium oxide	.58
Phosphorous pentoxide	1.44
Manganese oxide	trace
Loss on ignition	11.93
	100.24

This material was to be mixed in such proportion as to make a sound, strong Portland Cement. The composition of Portland Cement cannot vary within wide limits and certain ingredients like magnesia, and sulphuric anhydride must be low. The limit on magnesia is usually placed at 4 per cent in cement and that of sulphuric anhydride at 1.75 per cent. As regards the absence of both of these harmful ingredients the raw material is especially favored.

The necessary constituents of Portland Cement are silica, alumina and lime. According to Le-Chatelier, Portland cement clinker consists of alit, considered to be tri-calcium silicate $3CaO, SiO_2$ which is the active hardening element, and celit, considered to be tri-calcic aluminate,— $3CaO, Al_2O_3$ also setting and hardening in water. There are also present in small amounts calcium ferrate and mono, and dicalcium silicates.

Richardson states that the components of cement are present in the clinker as two solid solutions, alit— a solution of tricalcic aluminate in tri-calcic silicate and celit, a solution of dicalcic aluminate in di-calcic

silicate. The ratio of alit to celit may vary from 3 to 6 to 1.

According to Bleininger* the limits of composition of Portland Cement are as follows:

Silica	19 to 26 per cent
Alumina	4 to 11
Ferric oxide	2 to 5
Lime	58 to 67
Magnesia	0 to 5
Sulphuric anhydride	0 to 2.5
Alkalies	0 to 3.0

The following are typical cements analyses.**

	Lehigh Co., Penn.	Yankton, S. D.	Coldwater, Mich.
Silica	22.68	19.50	21.22
Alumina	6.70	7.70	7.51
Ferric oxide	2.35	4.30	3.83
Lime	62.30	60.00	63.75
Magnecia	3.41	0.80	0.82
Sulphuric anhydride	1.88	2.80	1.58
Alkalies	....	1.20	.....
Loss on ignition.	....	.....	1.02

According to the constitution of the cement there must be a definite ratio between lime, silica and alumina which for American cements may be expressed by the following empirical formula:

$$\text{Lime} = \text{Silica} \times 2.7 + \text{alumina} \times 1.$$

Many cements come quite close to this composition though more generally the lime falls somewhat below the figure obtained by the calculation.

The dried raw materials, the compositions of which have been mentioned were mixed in the proportion of 1 part shale to 3.30 parts limestone, after considerable experimentation with somewhat different mixtures, this proportion yielding the best result. Ac-

*Manufacture of Portland Cements. Bul. No. 3, Ohio Geol. Survey.

**From Taylor's Practical Cement Testing.

cording to calculation from analysis this mixture should burn to a cement of the following composition:

Silica	22.61 per cent
Alumina	7.43
Ferric oxide	1.60
Lime	65.51
Magnesia	1.07

The actual composition of the cement (sample No. 13) is as follows:

Silica	23.08 per cent
Alumnia	7.12
Ferric oxide	2.07
Magnesia	1.08
Lime	64.80
Sulphuric anhydride	0.66
Potassium oxide	0.52
Sodium oxide	0.21
Titanium oxide	0.26
Phosphorous pentoxide	0.53
Manganous oxide	trace
Carbonic oxide	trace
Total	100.33 ....

In the burning curves shown in Fix. 11 this cement is represented by curves 9, 12 and 13. Curves 7 and 8 are those of a cement made of 1 part of shale, and 3.5 parts limestone and 0.04 parts iron ore containing 80 per cent of ferric oxide, this last being added to slightly increase the iron content of the cement. This cement has the following composition:

Silica	22.04 per cent
Alumina	8.05
Ferric oxide	2.67
Lime	65.53
Carbonic acid	.016

The Preparation of the Raw Material.—The limestone and shale were crushed in a small Gates breaker (laboratory size) to a one quarter inch ring, and dried at approximately 100° C. Each constituent was then

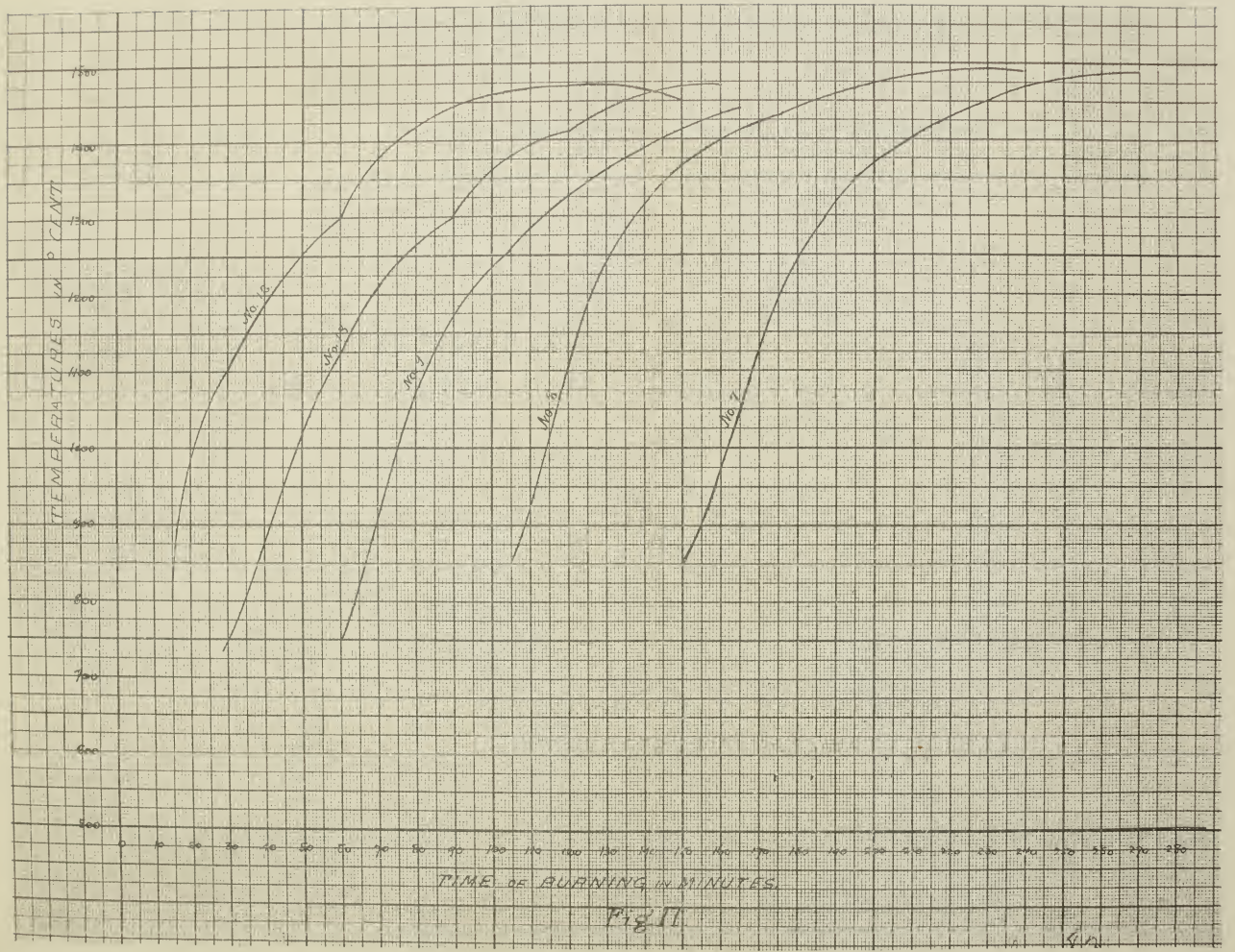

TEMPERATURES IN D CENT
1500
1300
1200
1100
1000
900
800
700
600
500
No. 10
No. 15
No. 1
No. 5
No. 7
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250
TIME OF BURNING IN MINUTES.

Fig 11

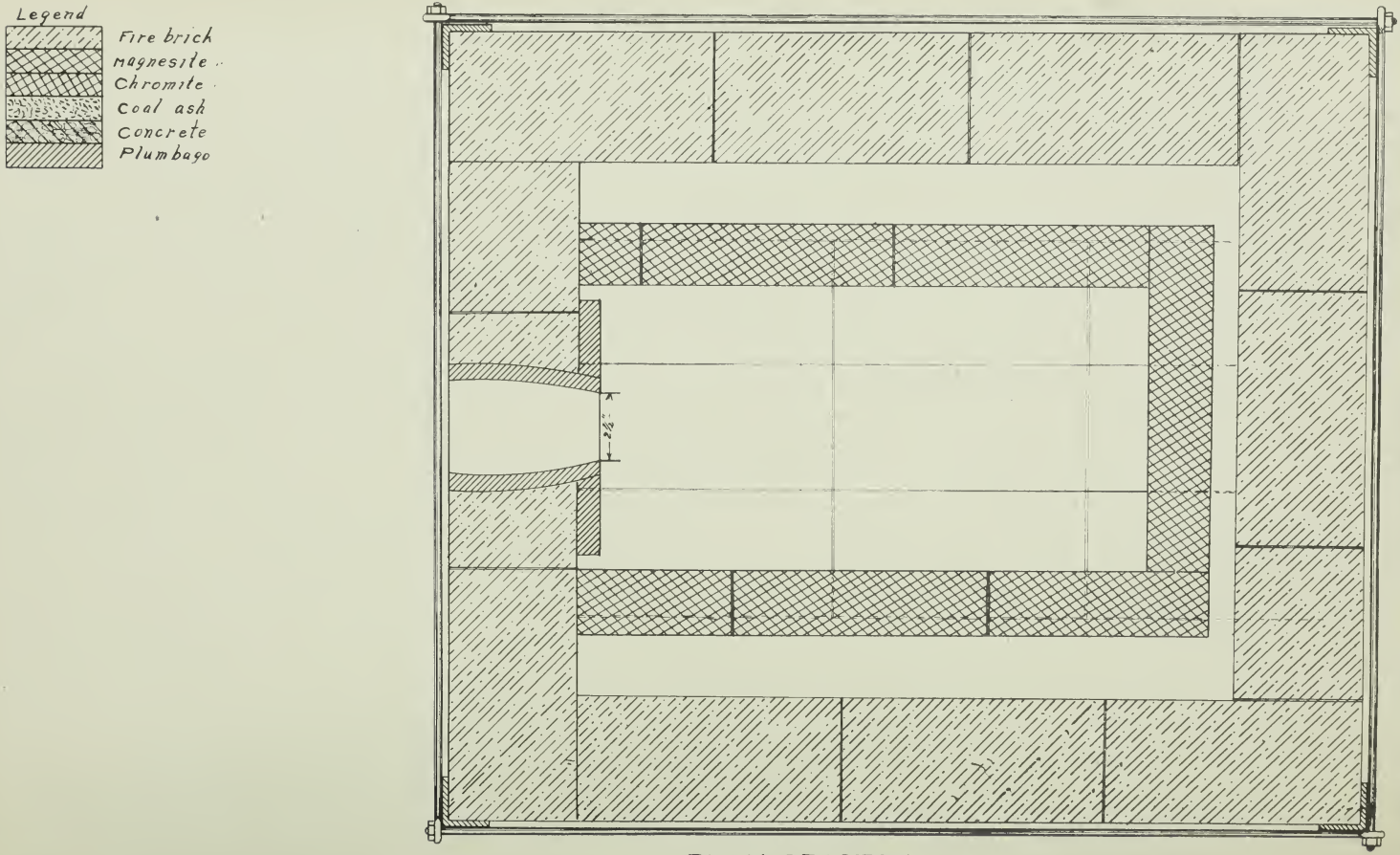
weighed out accurately in the proper proportion and ground by a Braun disk grinder so that all of it passed a 150 mesh laboratory screen. The ground products were then thoroughly incorporated by mixing on rubber cloth, after which in most instances the whole was again put through the grinder to more thoroughly incorporate it. Grinding the raw material together in the pulverizer was not very satisfactory as the more brittle limestone would grind first. The mass was then moistened with water until a ball of it when squeezed in the hand, would just hold together, and briquetted in a foot power cupel machine, to briquettes 1.25 in. in diamater and 1 in. deep. After drying to expel all moisture, these briquettes were ready for burning. A batch of material usually weighed 2200 grams, 500 grams of shale being taken as the unit.

Cement Burning. For the proper burning of cement a high temperature is necessary as the usual temperature required is between 1400° and 1500° C. The precise temperature depends upon the composition of the cement mixture and to some extent upon the length of time consumed in burning. Cement mixtures high in alumina, silica, or ferric oxide require a lesser temperature for combination of the constituents than cements high in lime. Highly aluminous cements are, however, undesirable as they usually prove defective. The amount of ferric oxide in a cement is largely incidental and determined by the composition of the raw materials and cannot ordinarily be varied without disturbing the important relation of silica to lime and to alumina. In the material used in the described experiments the limestone is very pure, and carries practically lime only, while the shale contains the alumina and silica, so that a variation in the alumnai also includes one of the silica contents of the cement.

The briquettes after burning make cement "clinker." The appearance of the clinker is an indication of the proper burning of the cement, and also

indicates roughly if the composition is approximately corret. Good clinker should be hard, but brittle, somewhat porous, but of sufficient specific gravity. The specific gravity of the burnt cement is the best general indication of proper burning. Depending somewhat upon composition the specific gravity of the ground cement should be from 3.10 to 3.20. These figures refer to cement that has been stored for some weeks. If the specific gravity is taken just afer burning it will range from 3.15 to 3.25. Taking the figures for stored cement a gravity below 3.10 indicates unburning and one above 3.20, overburning. The clinker from cements high in lime are often rather light and porous, and that from cements high in alumina, dense and compact, relatively speaking. The last mentioned kind of clinker is very apt to "dust" when taken from the furnace, e. g., to fall apart into powder on cooling.

The Experimental Furnace.—The furnace for the burning of the cement mixture should be so constructed that a temperature of 1500° C., can be obtained with it. The generating of this temperature in *any considerable* space for a prolonged time is attended with some difficulties. The first attempts were made with a small gasoline fired assay crucible furnace lined with fire clay tiling and bound with sheet iron. With a 2 inch Carey burner and gasoline at 20 lbs. pressure a temperature of 1400° C was obtained in the focus of the burner flame, i. e. only within a small space. Only a few briquettes could be clinkered in this way while the others in the furnace would be much underburned. Another difficulty encountered was that where the cement briquettes were in contact with the furnace lining they absorbed silica and alumina, thus destroying the original composition of the mixture, and causing them to "dust" or slough away on cooling. The attempt to burn the briquettes in a pot furnace in direct contact with coke proved unsatisfactory for experimental purposes as the amount of ash absorbed by the



PLAN SECTION

Fig. 10.

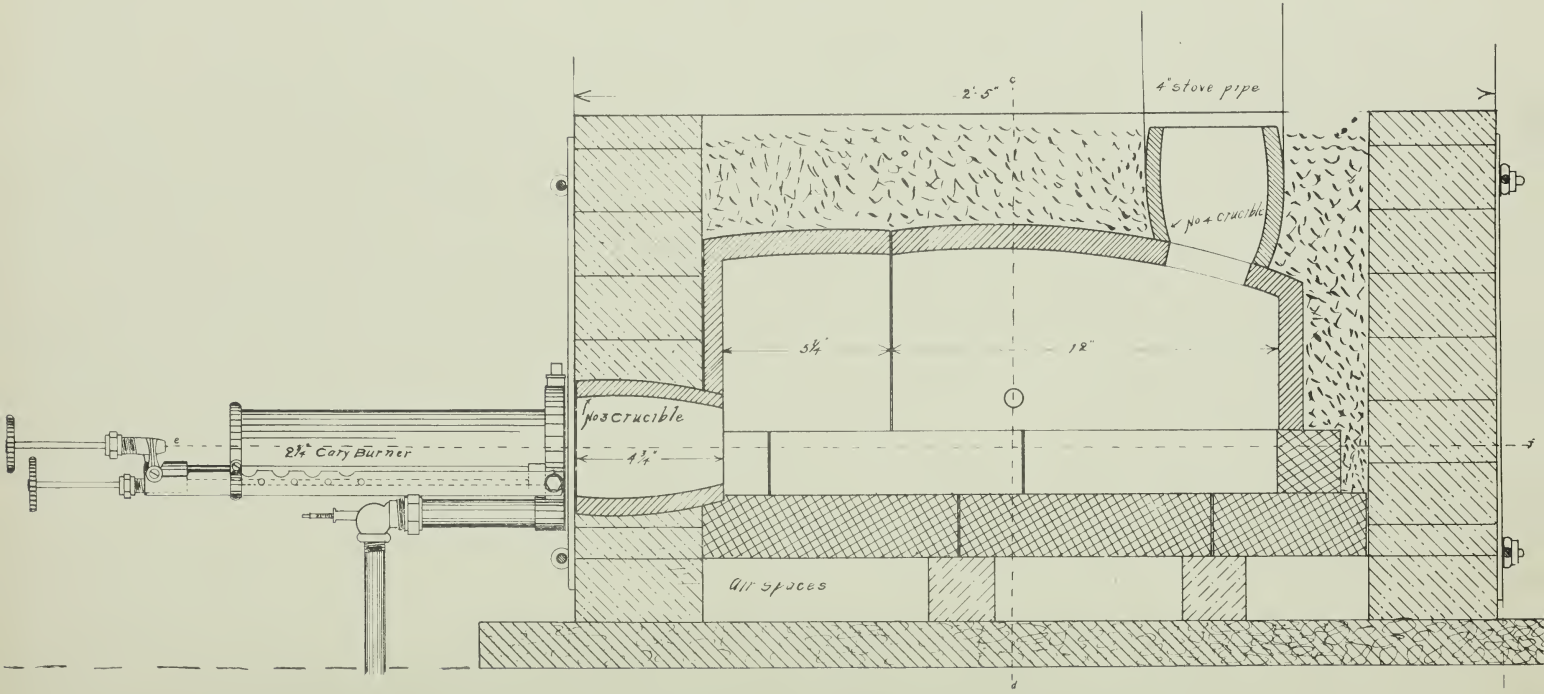
briquettes was such as to cause indefinite results. As a last sesort the following furnace was constructed. A graphite crucible was cut lengthwise below the median line and the larger half used. A flue opening was made at the top near the closed end. Thoroughly burnt coal ashes were spread on a cement top table and were confined by a rectangle of fire brick. A layer of chromite brick (a neutral refractory substance) was placed on the ashes and formed the bottom of the furnace. On this was placed the improvised graphite muffle as shown in the accompanying drawings. The outside walls of the furnace were laid up of fire brick in a mortar of fire clay and cement. The space between the walls of the furnace and the muffle was filled with ashes as shown in the drawing.

The flue was connected by pipe to the chimney. The only opening into the furnace was through the "burner boss" which was made of a section of graphite crucible. Whenever the furnace was charged or discharged the burner boss was removed, giving an ample cylindrical opening into the muffle, flush with the chromite bottom. The firing was done with a 2 in. Carey burner, with gasoline as fuel.

The burner was so arranged as to swing readily away from the burner opening and leave free access to the furnace. A porcelain pyrometer tube reaching to nearly the center of the furnace was built into the one side wall and afforded the means of obtaining the thermo-couple readings of "burning." Gasoline was burnt under a pressure of 50 to 60 lbs. per sq. in. In order to burn the quantity of gasoline implied by this pressure in a 2 in. burner, the draft in the furnace had to be considerable. This was obtained by the flue connections mentioned above. A uniform temperature throughout the muffle, of nearly 1500° C could be readily obtained, and batches of cement of 2000 grams, completely and readily clinkered in from 1½ to 2½ hours. The cement briquettes never ad-

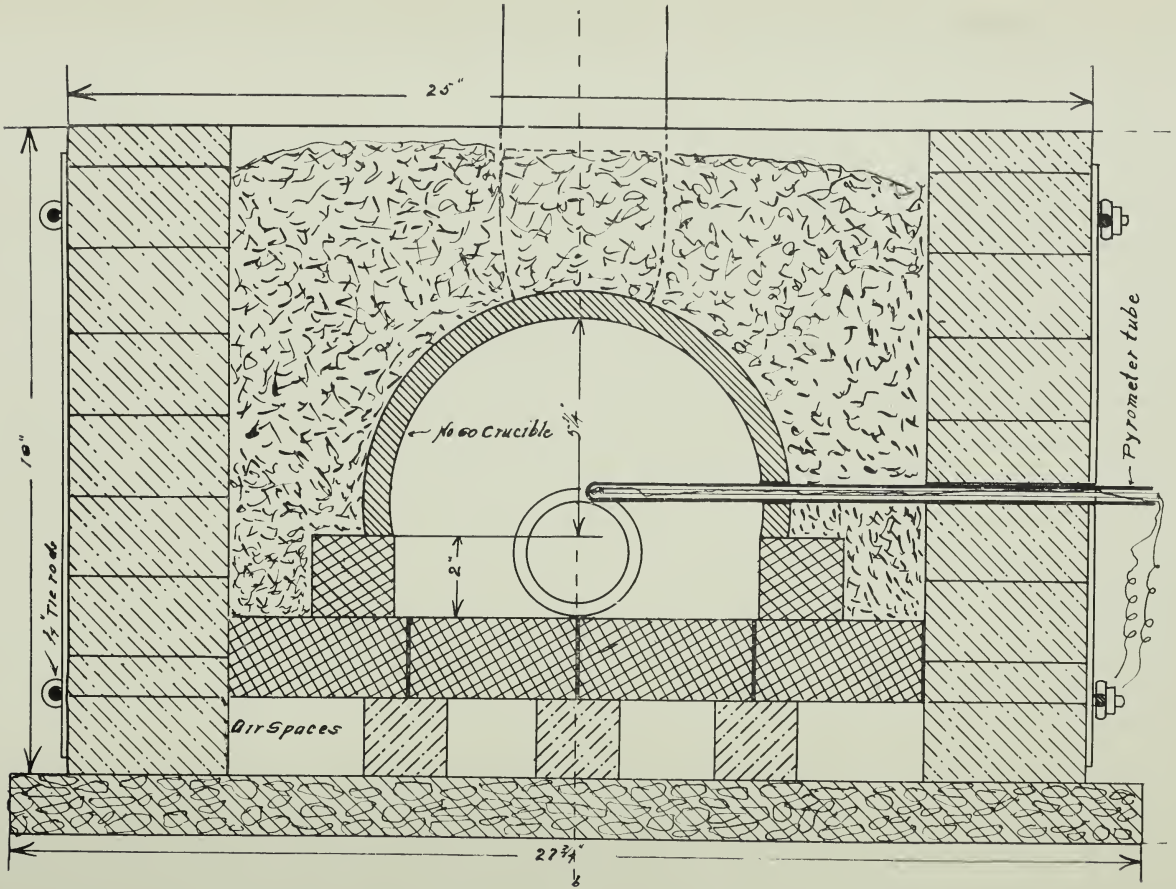
hered to the bottom of the furnace, and came out perfectly clean.

Preparation of the Cement for Testing.—The cement clinker was crushed and ground in the Braun grinder, to pass a 150 mesh screen thoroughly mixed and was ready for testing. Some samples of it were stored before testing, others, tested without storing.



LONGITUDINAL SECTION

Fig. 8.



CROSS SECTION

Fig 2

PART IV.

EXPERIMENTAL TESTS ON BLACK HILLS CEMENT.

MARK EHLE, JR., E. M.

Testing.

MARK EHLE, JR., E. M.

In testing the Portland Cement manufactured at the State School of Mines the object was to determine its suitability as a constructive material, within limitations esablished by the American Institute of Civil Engineers, through a committee appointed by that body in 1903. To this end all methods of procedure were in accordance with the specifications by them adopted.

The properties examined and reported herein include the following:: (1) Color; (2) Specific Gravity; (3) Activity; (4) Soundness; (5) Strength.

Color:—The color of a cement powder indicates little or nothing relative to its cementitious value and is of importance only when the cement is to be used in connection with exposed portions of a structure, a uniform shade being desired. All the cement made at the School of Mines was of the greenish gray shade, characteristic of many commercial Portlands.

Specific Gravity—According to the reports of the committee above mentioned, the determination of the Specific Gravity of a cement powder gives the best and quickest test for underburning or adulteration; the Specific Gravity of an underburned or impure product being usually lower than for the normal article. In the case of the samples herein reported the main interest lies in a comparison with well established figures, which for standard brands vary from 3.10 to 3.25. The tests for Specific Gravity were made with

the aid of the simple apparatus designed by LeChatelier, and shown in the accompanying Fig. 1. It consists of a flask (D) having a capacity of 120 cubic centimeters (72.32 cu. in.), the neck of which has a diameter of about 9 millimeters (0.35 in) and is expanded into a bulb (C) about midway of its length. The volume of this bulb between two marks at (E) and (F) is just 20 cubic centimeters, (1.22 cu. in.) and graduations on the tube, reading to tenths of a cubic centimeter, are continued upward from (F). The small funnel (B) has a tube long enough to extend just below (F).

In determining the Specific Gravity of a cement powder, the flask is filled with kerosene (free from water, to the point (E). The sample, having been first thoroughly dried and brought to the temperature of the liquid in the flask, a portion equal to about 64 grams (2.25 oz.) is carefully weighed out and introduced into the flask through the funnel (B). The level of the liquid is thus raised to some division on the graduated neck and this reading added to 20 gives the total volume displaced by the powder.

The Specific Gravity is then obtained from the following formula:

$$\text{Specific Gravity} = \frac{\text{Weight of Cement}}{\text{Displaced Volume}}$$

During the test, the flask is kept immersed in water in the jar (A), in order to avoid variations in the temperature of the liquid it contains.

Activity.—By this term is meant the rate of hardening of a cement after being made, with water, into a paste and allowed to stand undisturbed. As the degree of plasticity of the paste very much effects the rate of hardening, it becomes necessary to mix all samples to a uniform or, as it is termed, normal consistency, in order to establish a basis from which to make observations. The test for activity therefore in-

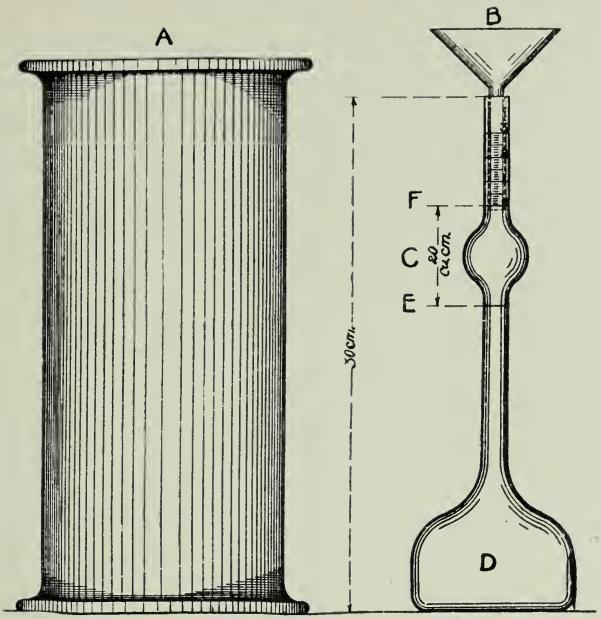


FIG. 1

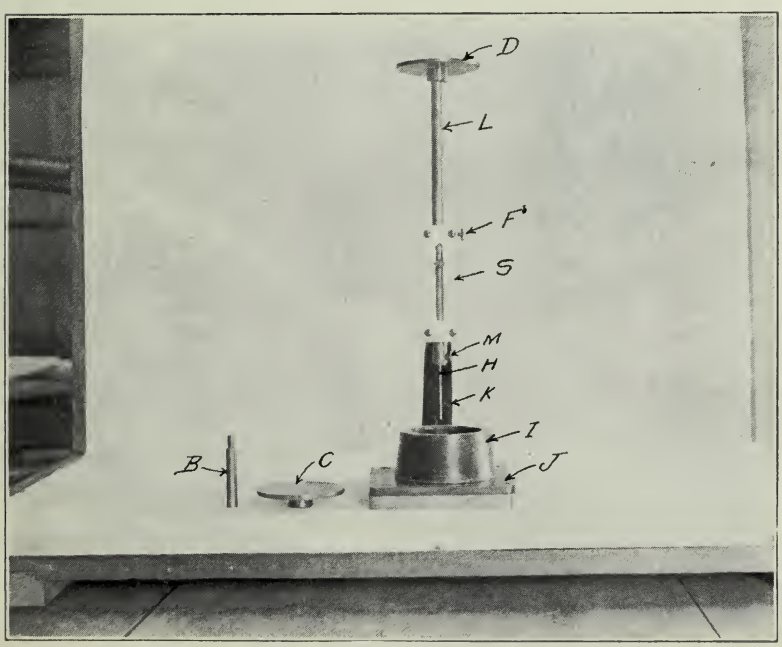


FIG. 2

cludes, for each sample, a preliminary test for normal consistency.

The methods employed in these determinations involved the use of the "Vicat Needle" apparatus its suitability as to constructive material, within limita shown in Fig. 2, and described as follows: (L) is a movable rod which may be adjusted vertically and held in any position by the set screw (F). An indicator attached to this rod moves over the scale (S) which is graduated to millimeters and attached to the frame (K). Into the lower end of the rod may be inserted either the cylinder (B), 1 centimeter in diamter or the needle (H) one millimeter in diameter, the set screw (M) securing either piece in place. Two interchangeable caps, (C) and (D) of different weights are provided and suitably marked, one for use in connection with the cylinder and the other with the needle; the combination of cap, rod and needle, assembled as shown, weighing just 300 gramms (10.58 oz.) which is also the weight of the combined cap, rod and cylinder. A conical rubber ring (1), 7 centimeters (2.76 in.) in diameter at the base and 4 centimeters (1.58 in.) high rests on the glass plate (J) and retains the paste which is to be tested.

In making the determination for normal consistency, about 500 gramms (17.58 oz.) of the cement powder are quickly worked into a thick paste, formed into a ball with the hands and pressed into the rubber ring through the larger opening, smoothed off and the face covered with the glass plate; plate and ring are then turned over and the smaller opening smoothed off with a spatula or trowel. The paste confined in the ring is then placed under the rod, bearing the cylinder and corresponding cap, the bottom of the cylinder being brought just into contact with the surface of the paste and quickly released. By definition the paste has a normal consistency when the cylinder

penetrates the mass to a depth of 10 millimeters (0.39 in).

These trial pastes are made up with varying percentages of water until the proper consistency is obtained. In making the test for activity the cylinder (B) is replaced by the needle (H), and the cap (D) replaces (A). The ring is then filled with paste of normal consistency, carefully smoothed off, and placed under the rod. At lengthening intervals of time the needle is brought just into contact with some point of the surface and quickly released. The setting is said to have commenced when the needle ceases to pass a point 5 millimeters (0.20 in.) above the upper surface of the glass and is said to have terminated the moment the needle does not visibly sink into the mass. The two points thus observed are termed the initial and final set, respectively, and the time of each period is to be noted from the instant of first addtion of water to the mixture. The time of initial set varies widely in different cements, ranging from 15 minutes to 12 hours and if of great importance in that it marks the time after which no internal disturbance of the mass should occur.

The final set marks the point when real hardening has commenced which hardening process may not be complete for years. The test pieces should in all cases be kept damp or immersed in water during the period of testing.

Soundness.—By this term is meant the ability of the cement to retain its strength and form indefinitely. Any tendency to swell, crack or disintegrate, after the mass has set, indicates failure in his respect.

As unsoundness is frequently caused by the presence in the cement of some active agent such as free lime or magnesia and as the evil effects of these elements would, under normal conditions, not be manifest for a considerable period, it becomes desirable to accelerate their action and by thus noting the behavior of the cement over a relatively short period, under

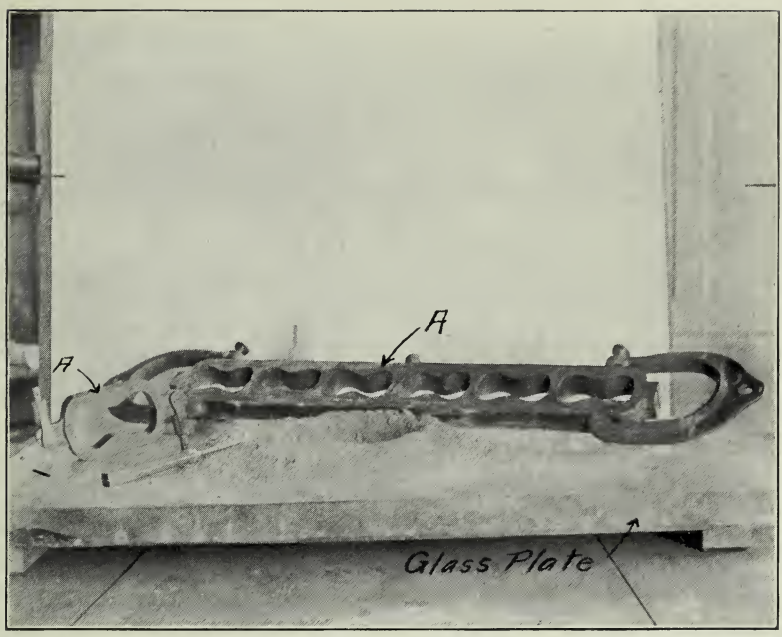


FIG. 3

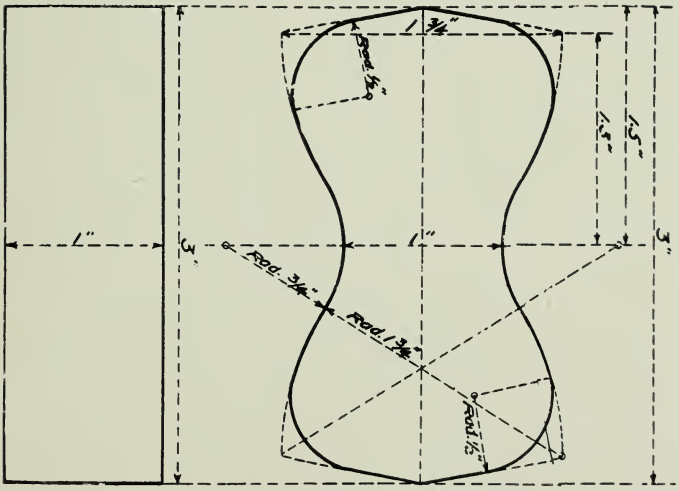


FIG 4

abnormal conditions, determine what would probably be their action over a long period under normal conditions.

This accelerating action is usually effected by the use of heat, the test being made as follows. From a paste of normal consistency a number of lens shaped patties are moulded on watch glasses, each sample being smoothed out to a thin edge around its circumference. These patties are then set aside in a moist atmosphere until quite hard. These samples, still adhering to the glasses, are then placed in a vessel of water, the temperature of which is gradually raised to the boiling point and maintained there for at least three hours. To pass a satisfactory test the patties should remain intact without any sign of cracking, disintegration or distortion.

Strength.—The tests for strength were made by submitting moulded specimens, having a minimum cross sectional area of one square inch, to a tensile stress sufficient to rupture them, and noting the magnitude of such stress, for the purpose of comparison. On account of the lack of suitable apparatus, no tests of crushing strength were made. Extensive investigation has shown, however, that the resistance in compression may be assumed as about ten times that in tension.

In the case of each batch of cement burned, tests were made on specimens made from the pure or "neat" cement and also on specimens of mortar, composed of certain percentages of cement and standard sand. By standard sand is meant a clean, sharp sand, all of which will pass a twenty mesh and remain on a thirty mesh screen. The effect of age was also considered, specimens being tested at seven and also at twenty-eight days from time of mixing.

The form of mould used in making the briquettes or test pieces is shown at (A), Fig. 3, the samples being retained in the moulds, in a damp atmosphere, until hard, when they were removed and kept under water

until tested. Fig. 4 shows the feature of one of these standard briquettes.

The machine used in making these tests, the property of the School of Mines, is the standard type manufactured by Riehle Bros. of Philadelphia, Pa., and has a capacity of 2000 lbs. It is shown in Fig. 5. In operating, the briquette to be tested is introduced into the clips or jaws at (A) (also shown in detail in Fig. 6). By means of a small hand wheel operating the worm gear at (C), a gradually increasing tensile stress is brought to bear on the specimen, this stress being counterbalanced, through a compound lever, by the rider (R) running on the graduated beam (D), the position of the rider being controlled by the small hand wheel (W). and exact equilibrium being at all times maintained by observing the pointer (P). The stress is increased slowly and uniformly until rupture of the piece occurs, the stress producing it being then read directly, in pounds, from the position of the rider on the beam.

Results.—In all, some thirteen batches of cement were burned and tested, the results obtained from five of them being shown in the following tabular view.

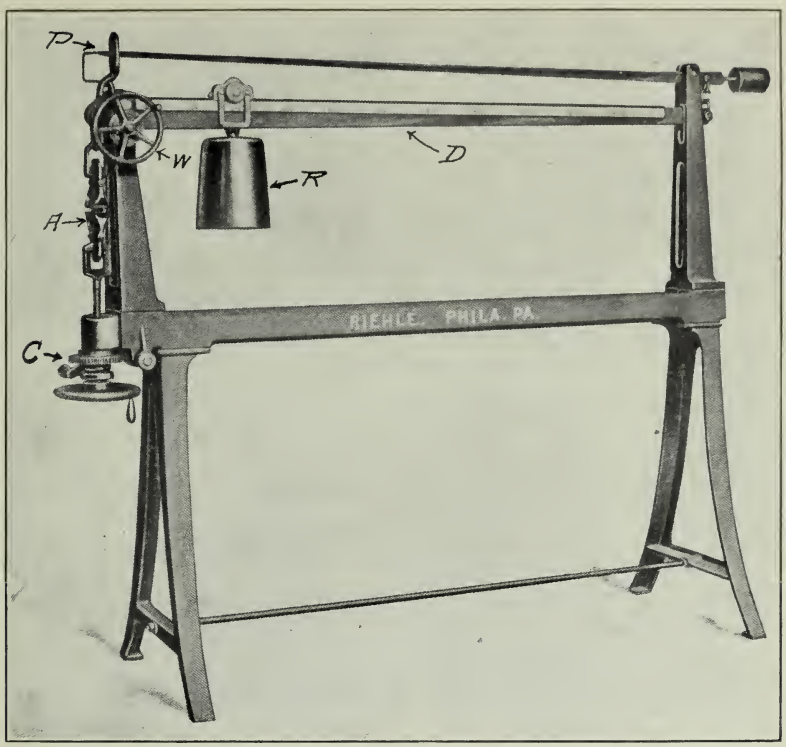
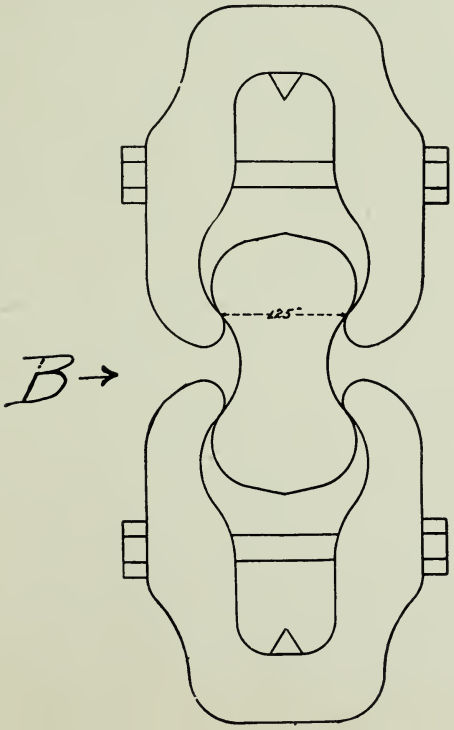


FIG. 5



Number of Batch of Cement	Number of Sample	Specific Gravity	Activity or Time of Initial Set	Soundness	Strength in Tension			
					After 7 Days		After 28 Days	
					Neat	3 Pts. Sand 1 Pt. Cem't	Neat	3 Pts. Sand 1 Pt. Cem't
7	4	3.18	11 hours	Perfect	414			
"	6	"	"	"	413			
"	5	"	"	"			540	
"	1	"	"	"			555	
8	1	3.20	10 hours	Perfect	355			
"	3	"	"	"	360			
"	7	"	"	"		145		
"	10	"	"	"		96		
"	5	"	"	"			570	
"	6	"	"	"			592	
"	11	"	"	"				178
"	12	"	"	"				191
9	1	3.25	9 hours	Perfect	459			
"	3	"	"	"	422			
"	10	"	"	"		116		
"	11	"	"	"		107		
"	4	"	"	"			543	
"	2	"	"	"			572	
"	14	"	"	"				191
"	15	"	"	"				205
12	3	3.17	10 hours	Perfect	597			
"	4	"	"	"	558			
"	5	"	"	"		171		
"	6	"	"	"		179		
"	1	"	"	"			560	
"	2	"	"	"			510	
"	7	"	"	"				218
"	8	"	"	"				285
13	1	3.16	10 hours	Perfect	358			
"	2	"	"	"	413			
"	6	"	"	"		164		
"	7	"	"	"		168		
"	3	"	"	"			565	
"	4	"	"	"			670	

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